

## **REMARKS**

Claims 1-23 are rejected. Claims 1, 7, 8, and 22 have been amended. Claims 6, 12, and 15 have been canceled. Claims 1-5, 7-11, 13, 14, and 16-23 are presently pending in the application. Favorable reconsideration of the application in view of the following remarks is respectfully requested.

The basis for the amendment of claim 1 is original claims 1, 6, 12, and 15 and pg. 6, lines 9-11 of the specification as originally filed. The basis for the amendment of claim 22 is claim 23 as originally filed.

### **Specification:**

The status of the copending application cited on page 1 of the specification has been updated, as requested by the Examiner.

### **Rejection of Claim 22 under 35 USC § 112:**

The Examiner has rejected Claim 22 under 35 U.S.C. § 112, second paragraph, as being indefinite for failing to particularly point out and distinctly claim the subject matter which applicants regard as the invention, indicating that the term "support enhanced by at least" in claim 22 is indefinite. Claim 22 has been amended to overcome this rejection.

### **Rejection of Claims 1-3 and 5-23 Under 35 U.S.C. §102(b):**

The Examiner has rejected Claims 1-3 and 5-23 under 35 U.S.C. § 102(b) as being fully met by Christian et al, which discloses imaging elements with antistatic layers with swellable clay particles comprising natural clay intercalated or exfoliated with hydrophilic polymers.

Christian discloses an imaging element which includes a support, an image-forming layer superposed on the support, a transparent magnetic recording layer superposed on the support; and an electrically-conductive layer superposed on the support. The transparent magnetic recording layer is composed of magnetic particles dispersed in a first film-forming polymeric binder. The electrically-conductive layer includes electrically-conductive metal-containing colloidal particles, swellable, smectite clay particles, a first polymeric binder which can sufficiently intercalate inside or exfoliate the smectite clay particles and a second film-forming polymeric binder, wherein the electrically-conductive metal-containing particles and the polymer-intercalated or polymer-exfoliated smectite clay particles are dispersed for use in photographic and thermally-processable imaging elements.

The present invention relates to imaging elements having improved mechanical properties as a result of incorporation of a natural clay-containing layer. These imaging elements are characterized by a support, an imaging layer, and at least one layer comprising a clay nanocomposite wherein said nanocomposite comprises a hydrophilic organic splayant and at least one natural clay particle having an aspect ratio of from 100:1 to 400:1 and a length greater than 0 and less than or equal to 700 nm (0.7 $\mu$ m).

A claim is anticipated under 102(b) only if each and every element as set forth in the claim is found, either expressly or inherently, in a single prior art reference. Verdegaal Bros. V. Union Oil Co. of California, 814 F.2d 628, 631, 2 USPQ2d 1051, 1053 (Fed. Cir. 1987). Applicants have amended claim 1 to incorporate an aspect ratio range not specified in Christian that is, an aspect ratio of from 100:1 to 400:1. The aspect ratio according to Christian is from 25:1 to 50:1 (col. 8, lines 20-21). As a result, Christian fails to the elements of the presently amended claims and fails to anticipate the presently claimed invention. Therefore, the Applicants request that the Examiner reconsider and withdraw the rejection.

**Rejection of Claims 1-8 and 10-23 Under 35 U.S.C. §102(b):**

Claims 1-8 and 10-23 are rejected under 35 U.S.C. § 102(b) as being fully met by Aono. Aono (see particularly column 3, line 30 - column 4, line 25; column 5, lines 8-16; column 23, lines 52-60) disclose imaging elements with layers comprising natural clay particles in hydrophilic polymers, preferably gelatin, which are splayant materials. The clay particles of instant claims 1-8 and 11-23 are not required to be intercalated or exfoliated. Also, the clay particles in Aono are disclosed as exfoliated, i.e. the plates or layers of the clay particles are cleaved into a sol.

Aono discloses a silver halide photographic material which has a layer containing a swellable inorganic stratifying compound. The photographic material or a photographic element has at least one light-sensitive silver halide emulsion layer which comprises a swellable inorganic stratifying compound and may also contain at least one light-insensitive layer containing a swellable inorganic stratifying compound. The photographic element may also comprise a support having thereon a light-sensitive element comprising at least one light-sensitive silver halide emulsion layer and an image-receiving element, wherein at

least one layer of said photographic element comprises a swellable inorganic stratifying compound and which may also have a light-insensitive layer containing the swellable material. The element may also comprise a support having thereon a light-sensitive element comprising at least one light-sensitive silver halide emulsion layer and a processing element which is capable of being closely contacted with said light-sensitive element during development, wherein a swellable inorganic stratifying compound is present in either the photographic element or at least one light-insensitive layer.

The present invention relates to imaging elements having improved mechanical properties as a result of incorporation of a natural clay-containing layer. These imaging elements are characterized by a support, an imaging layer, and at least one layer comprising a clay nanocomposite wherein said nanocomposite comprises a hydrophilic organic splayant and at least one natural clay particle having an aspect ratio of from 100:1 to 400:1 and a length greater than 0 and less than or equal to 700 nm (0.7 $\mu$ m).

A claim is anticipated under 102(b) only if each and every element as set forth in the claim is found, either expressly or inherently, in a single prior art reference. Verdegaal Bros. V. Union Oil Co. of California, 814 F.2d 628, 631, 2 USPQ2d 1051, 1053 (Fed. Cir. 1987). Applicants have amended claim 1 to incorporate clay particles having a length greater than 0 and less than or equal to 700 nm (0.7 $\mu$ m). Aono disclose particles of 1-20  $\mu$ m (col.3, lines60-61). As a result, Aono fails to the elements of the presently amended claims and fails to anticipate the presently claimed invention. Therefore, the Applicants request that the Examiner reconsider and withdraw the rejection.

**Rejection of Claims 1-3, 5, 6 and 9-23 Under 35 U.S.C. §102(e):**

Claims 1-3, 5, 6 and 9-23 are rejected under 35 U.S.C. § 102(e) as being anticipated by Majumdar et al. '696. The applied reference has a common assignee with the instant application. Based upon the earlier effective U.S. filing date of the reference, it constitutes prior art under 35 U.S.C. § 102(e). This rejection under 35 U.S.C. § 102(e) might be overcome either by a showing under 37 CFR 1.132 that any invention disclosed but not claimed in the reference was derived from the inventor of this application and is thus not the invention "by another," or by an appropriate showing under 37 CFR 1.131. Majumdar et al. '696 (see particularly column 2, lines 21-28; column 3, lines 5-27; column 3, line

65 - column 5, line ,8; column 10, lines 48-66; column 11, lines 54-65; Example 1) disclose imaging elements with support layers with layers of nanocomposite particles for stiffer supports. The nanocomposite particles include natural clay intercalated and/or exfoliated with splayants. The layers with clay particles may be coated on the top or bottom of a support to increase stiffness.

Majumdar discloses an imaging member comprising an image layer and a support comprising at least one layer comprising an inorganic particle having an aspect ratio of at least 10 to 1, a lateral dimension of between 0.01  $\mu\text{m}$  and 5  $\mu\text{m}$ , and a vertical dimension between 0.5 nm and 10 nm, and polymeric resin for use as an improved base for photographic materials.

The present invention relates to imaging elements having improved mechanical properties as a result of incorporation of a natural clay-containing layer. These imaging elements are characterized by a support, an imaging layer, and at least one layer comprising a clay nanocomposite wherein said nanocomposite comprises a hydrophilic organic splayant and at least one natural clay particle having an aspect ratio of from 100:1 to 400:1 and a length greater than 0 and less than or equal to 700 nm (0.7 $\mu\text{m}$ ).

A claim is anticipated under 102(e) only if each and every element as set forth in the claim is found, either expressly or inherently, in a single prior art reference. Verdegaal Bros. V. Union Oil Co. of California, 814 F.2d 628, 631, 2 USPQ2d 1051, 1053 (Fed. Cir. 1987). Applicants have amended claim 1 to incorporate a hydrophilic organic splayant. Majumdar fails to mention the use of gelatin or hydrophilic organic splayants to splay the particles as presently claimed. As a result, Aono fails to the elements of the presently amended claims and fails to anticipate the presently claimed invention. Therefore, the Applicants request that the Examiner reconsider and withdraw the rejection.

**Rejection of Claims 1-3, and 5-23 Under 35 U.S.C. §102(e):**

Claims 1-3 and 5-23 are rejected under 35 U.S.C. § 102(e) as being anticipated by Dontula et al. The applied reference has a common assignee with the instant application. Based upon the earlier effective U.S. filing date of the reference, it constitutes prior art under 35 U.S.C. § 102(e). This rejection under 35 U.S.C. § 102(e) might be overcome either by a showing under 37 CFR 1.132 that any invention disclosed but not claimed in the reference was derived from the inventor of this application and is thus not the invention "by another," or by an

appropriate showing under 37 CFR 1.131. Dontula et al. (see particularly column 4, lines 1-15; column 6, lines 44-57; column 7, line 65 - column 8, line 20; column 9, line 63 - column 10, line 60; column 11, line 44 - column 12, line 15; Example 1) disclose imaging elements with supports comprising layers comprising clay nanocomposite materials of natural clay and splayant including polymers of instant claim 8. Example 1 uses the same nanocomposite clay material as in the Examples of applicants' specification.

Dontula discloses an imaging member comprising a duplitized imaging layer and a support comprising at least one layer comprising an inorganic particle having an aspect ratio of at least 10 to 1, a lateral dimension of from 0.01  $\mu\text{m}$  to 5  $\mu\text{m}$ , and a vertical dimension from 0.5 nm to 10 nm, and polymeric resin matrix to provide a novel translucent nanocomposite support for use in photographic reflection and transmission imaging applications.

The present invention relates to imaging elements having improved mechanical properties as a result of incorporation of a natural clay-containing layer. These imaging elements are characterized by a support, an imaging layer, and at least one layer comprising a clay nanocomposite wherein said nanocomposite comprises a hydrophilic organic splayant and at least one natural clay particle having an aspect ratio of from 100:1 to 400:1 and a length greater than 0 and less than or equal to 700 nm (0.7 $\mu\text{m}$ ).

A claim is anticipated under 102(e) only if each and every element as set forth in the claim is found, either expressly or inherently, in a single prior art reference. Verdegaal Bros. V. Union Oil Co. of California, 814 F.2d 628, 631, 2 USPQ2d 1051, 1053 (Fed. Cir. 1987). Applicants have amended claim 1 to incorporate a hydrophilic organic splayant. Dontula, col. 11, line 44-col.12, line 15 disclose the use of an amphiphilic copolymer, one part of which is compatible with the layered material, and one part compatible with the matrix material. Dontula does not disclose the use of a hydrophilic organic splayant capable of being compatible with both the layered material and the matrix. As a result, Dontula fails to the elements of the presently amended claims and fails to anticipate the presently claimed invention. Therefore, the Applicants request that the Examiner reconsider and withdraw the rejection.

**Rejection of Claims 1-23 Under 35 U.S.C. §102(e):**

The Examiner has rejected Claims 1-23 under 35 U.S.C. § 102(e) as being anticipated by Rao et al, but indicates that this rejection under 35 U.S.C. § 102(e) might be overcome either by a showing under 37 CFR 1.132 that any invention disclosed but not claimed in the reference was derived from the inventor of this application and is thus not the invention "by another," or by an appropriate showing under 37 CFR 1.131.

Rao discloses thermally developable materials include an imaging layer containing a non-photosensitive source of reducible silver ions. Disposed over the imaging layer is a barrier layer that comprises inorganic filler particles that are intercalated or exfoliated with a hydrophilic or water- dispersible polymer. The particles have a length to thickness ratio of from about 10 to about 1000. The barrier layer can prevent migration of diffusible imaging components and by-products resulting from high temperature imaging and/or development. These thermally developable materials include both thermographic and photothermographic materials.

The present invention relates to imaging elements having improved mechanical properties as a result of incorporation of a natural clay-containing layer. These imaging elements are characterized by a support, an imaging layer, and at least one layer comprising a clay nanocomposite wherein said nanocomposite comprises a hydrophilic organic splayant and at least one natural clay particle having an aspect ratio of from 100:1 to 400:1 and a length greater than 0 and less than or equal to 700 nm (0.7 $\mu$ m).

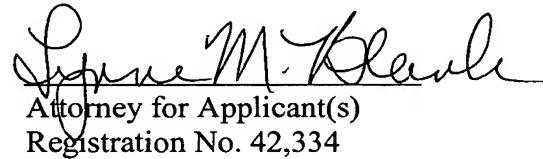
Under 35 USC 102(e), a person shall be entitled to a patent unless the invention was described in a patent granted on an application for patent by another filed in the United States before the invention thereof by the applicant for patent, or on an international application by another who has fulfilled the requirements of paragraphs (1), (2), and (4) of section 371(c) of this title before the invention thereof by the applicant for patent. When any claim of an application is rejected under 102(e), the inventor of the subject matter of the rejected claims may submit an appropriate oath or declaration to establish invention of the subject matter of the rejected claim prior to the effective date of the reference. The showing of facts shall be such, in character and weight, as to establish reduction to practice prior to the effective date of the reference, or

conception of the invention prior to the effective date of the reference coupled with due diligence from prior to said date to a subsequent reduction to practice or to the filing of the application. Original exhibits of drawings or records, or photocopies thereof, must accompany and form part of the affidavit or declaration or their absence satisfactorily explained.

The Applicants have attached an appropriate Declaration under 37 CFR 1.131, including photocopies of original Exhibits A-F, establishing that the reduction to practice of the subject matter of Claims 1-23 occurred prior to the effective date of Jan. 14, 2003 of the reference to Rao. Therefore, the Applicants request that the Examiner reconsider and withdraw the rejection.

It is believed that the foregoing is a complete response to the Office Action and that the claims are in condition for allowance. Favorable reconsideration and early passage to issue is therefore earnestly solicited.

Respectfully submitted,

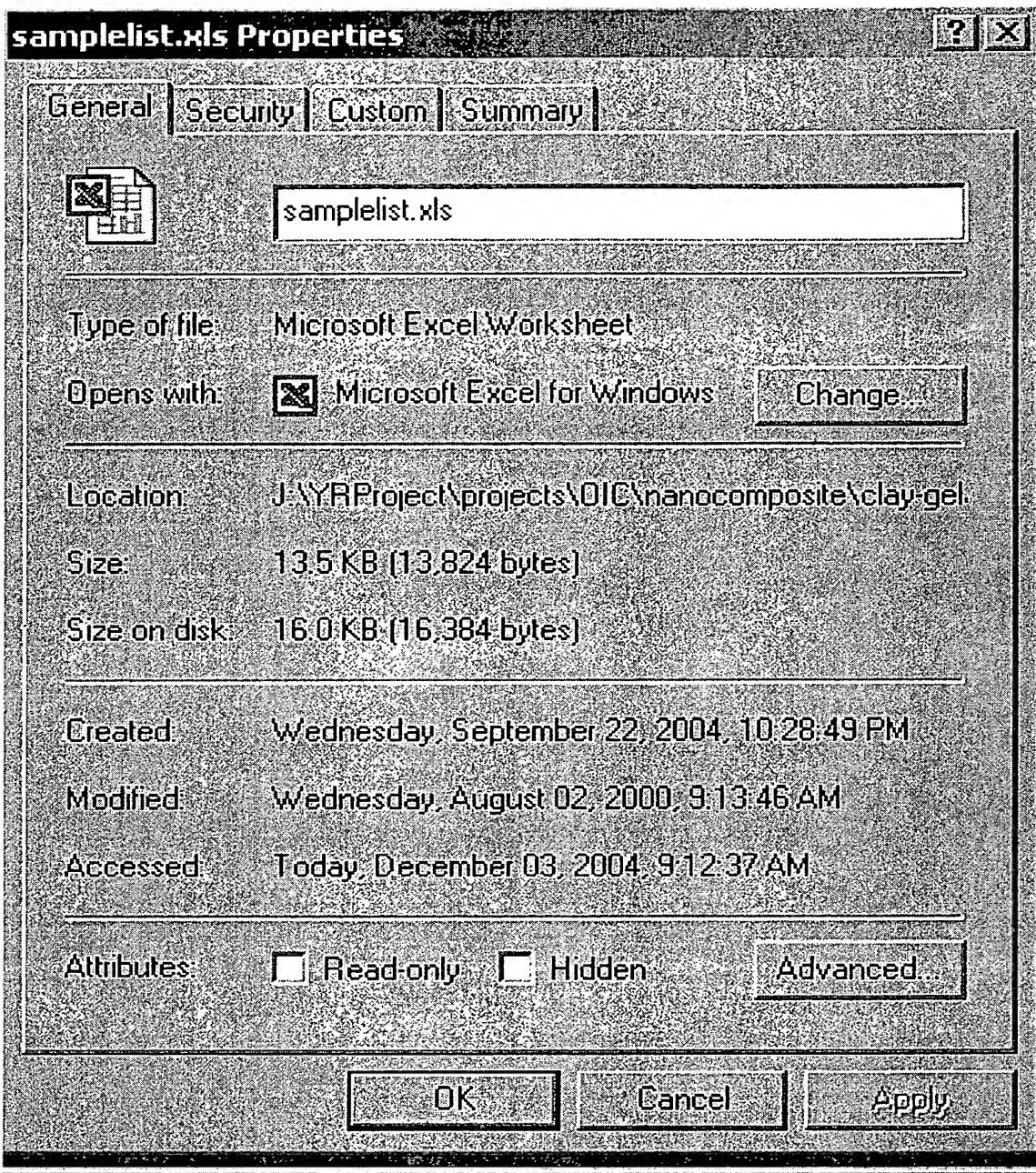


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If the Examiner is unable to reach the Applicant(s) Attorney at the telephone number provided, the Examiner is requested to communicate with Eastman Kodak Company Patent Operations at (585) 477-4656.

## samplelist.xls Properties



## EXHIBIT A

page 1 of 2 pages  
10/633,806

Sample list for scratch resistance

ID	Composition	Coating thickness mil
gel-7-7-2	pure gelatin	.35-1.15
5cloisitegel-7-7-b-5	5:95/cloisite:gelatin	.55-.7
5laponitegel-7-7-1c-6	5:95/laponite:gelatin	.25-1.55
10cloisitegel-7-7-b-9	10:90/cloisite:gelatin	.25-.8

Excel file dated : 8/2/2000

Filename: sample list.xls

Directory : project 1016 / nanocomposite / clay-gelatin/scratch

file property print out

**EXHIBIT A**  
page 2 of 2 pages  
10/633,806

Scratch under 5 gram using 3 mil stylus.doc Properties

?

X

General

Security

Custom

Summary



Scratch under 5 gram using 3 mil stylus.doc

Type of file: Microsoft Word Document

Opens with: Microsoft Word for Windows

[Change...](#)

Location: J:\YRPProject\projects\OIC\nanocomposite\clay-gel.

Size: 413 KB (422,912 bytes)

Size on disk: 416 KB (425,984 bytes)

Created: Wednesday, September 22, 2004 10:28:51 PM

Modified: Wednesday, August 16, 2000 9:13:56 AM

Accessed: Today, December 03, 2004 8:38:02 AM

Attributes:

Read-only

Hidden

[Advanced...](#)

[OK](#)

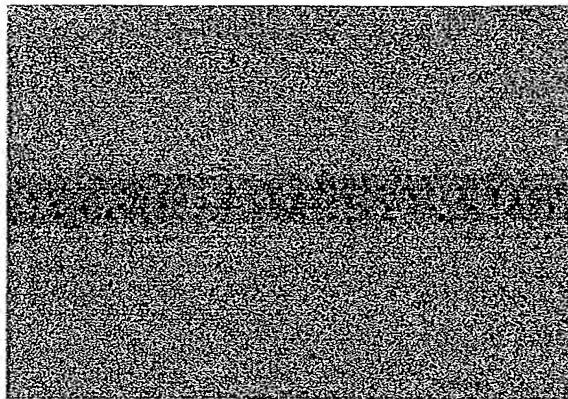
[Cancel](#)

[Apply](#)

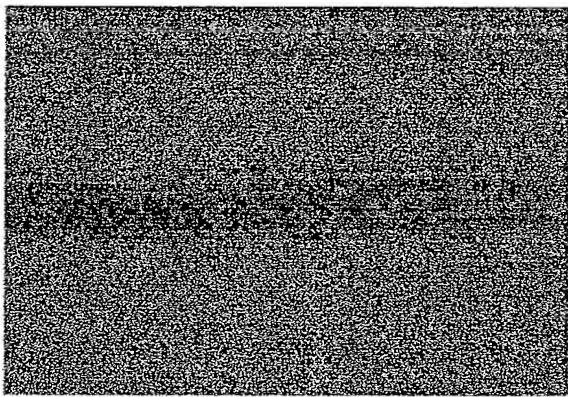
EXHIBIT B

page 1 of 3 pages

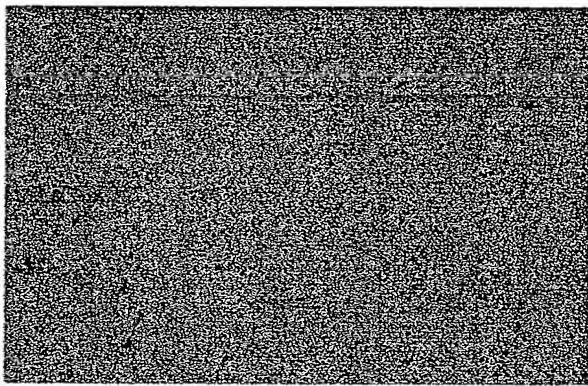
10/633,806



**Scratch of a gelatin film under 5 gram using 3 mil stylus**

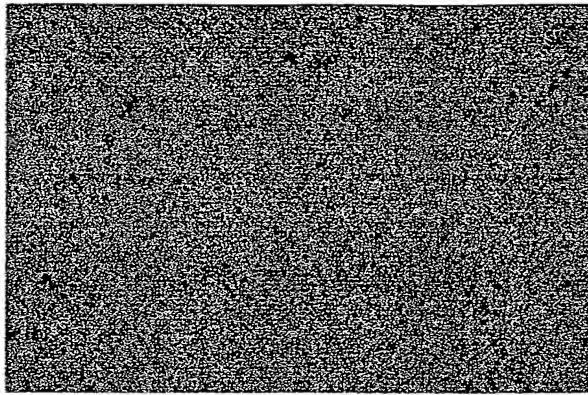


**Scratch of a 5:95/laponite:gelatin film under 5 gram using 3 mil stylus**



**Scratch of a 5:95/cloisite:gelatin film under 5 gram using 3 mil stylus**

**EXHIBIT B**  
page 2 of 3 pages  
10/633,806



Scratch of a 10:90/cloisite:gelatin film under 5 gram using 3 mil stylus

Excel File dated 8/16/2000

File name: scratch under 5 gram using 3 mil stylus .doc

Directory: project 101C/nanocomposite/clay-gelatin/scratch/  
pictures

**EXHIBIT B**  
page 3 of 3 pages  
10/633,806

Date 6-29-00, 7-7-00

Problem: Gelatin - clay. (Sample)

11.  $\approx$  clay size gel 627A2-1 uniform.(1)  $T = 0.75$  mil(2)  $T = 0.6$  mil, E.F(3)  $T = 0.65$  mil(4)  $T = 0.7$  mil
 $E = 579121 \text{ psi}$ ,  $\sigma = 14105$ ,  $\Sigma = 8.8\%$ ,  $T = 92.6$  (32.4)  
 (45951) (904), (2.4) psi

May: 15197 11.4 130.7

7-7-00.

10:30AM. Weigh Gelatin (30-122) 20 g

+ 480 g water Deion

→ 4% gel slurry

10:50AM. Put it in 50°C water bath mixing. (lightening mixing)  
— 11:10AM

11:00AM. Weigh Nanocor PEGV (PV-114-98)

10.17 g

11:40AM → + 240 g Deion water. (stumps first). RM mixing.

11:20AM Weigh SCP (apomite<sup>105</sup>) (13439-6/1244) 10.19 g

+ 240 g Deion water. Brownish slurry,

Put into 50°C bath. Lightening mixing.  
(translucent mixture)

First stir water to form Vortex, then add in clay.

11:40AM. Take 5g 4% clay size 6-1400.

5.05g. + 45g warm Deion water  
(50°C)EXHIBIT C  
page 1 of 6 pages  
10/633,806

Signature

The foregoing disclosed to me on

10/10/01

Witness

Date 7-7-00

m. 7-7-00 Gelatin - clay

12:00pm add 95 g gelatin 4% 6-7-00 (50°C)

40mA stir, small blade, ~50°C (Teflon container  
(No bubble))

12:10 PM Weigh 10.06g Cloisite - 6-14-00

+ 49.3g Deion (40°C) water

Weigh. 90g 4% gelatin 7-7-00

0.5 K rpm, (PE 400ml container).  
(Center bubble)

1:30pm Stop the 5% cloisite mixture (Temp. approaches 60°C.)

Weigh. total weight = 224.57g. Some bottom.

- O.W 108.09g after weighing. no bubble

Cone: 40ml

116.48g. seen.

1:40pm Weigh 5g Laponite RDS-7-7 (4%)

+ 42.28g Deion Water (55°C). Put in 55°C Cowles Mixer

1:50pm Weigh 95g 4% Gel shn. 40mA.

Add to Laponite mixture.

1:55pm 5 Cloisite gel - 7-7-B-1

130°F coating, set time 1 min., chill set 10s after reaching

low Temp. It's flowable during transportation.

(50°F)

2:00 pm. 5 Cloisite gel - 7-7-B-2.

120°F coating. set time 30s. chill set 10s.

Not flowable as the previous one

2:05pm. 5 Cloisite gel - 7-7-B-3

110°F coating. set time 1 min. chill set 10s

very flowable (compared to previous two, spilled some)

## EXHIBIT C

page 2 of 6 pages

10/633,806

Signature

B. J. M. Long

The foregoing disclosed to me on

B. J. M. Long

Signature

Witness

Book No. BB 9283

RESEARCH / DEVELOPMENT

EASTMAN KODAK COMPANY

7-7-00

Item: Gelatin - Clay

10pm. 5 Cloisitegel - 7-7-B-5.

120°F. On Ucoated PET 1min set time 105 chillset  
flowable (~B-3)

15pm 5 Cloisitegel - 7-7-B-4

120°F. On Bare PET. 5min. set time for chillset  
not flowable

0pm. Move Naucore PGV to 50°C Bath

? Need to check the total weight to make sure  
it's not contaminated by water.

2pm. Took 10 Cloisitegel mixture out.

top soaps. total weight = 148.42g.

- container weight = 26.17g

124.3g.

0pm. 10 Cloisitegel - 7-7-B-8

120°F. on Bare PET. 1min. set time. 105 chillset  
flowable, TABLE FLAT

3pm. 10 Cloisitegel - 7-7-B-9

ON Ucoated PET. 120°F. 1hr set time for chillset  
TABLE, Flat

4pm. gel - 7-7-1

4% gelatin sln. 120°F. 1min set time - 105 chillset  
ON Bare PET flowable table flat

5pm. gel - 7-7-2

4% gelatin sln. ON Ucoated PET. 120°F 1hr set time - 105 chillset  
very flowable. spill a little glass flat

5pm. gel - 7-7-3

EXHIBIT C

page 3 of 6 pages  
10/633,806

86 I.P.S.

Signature

The foregoing disclosed to me on 7-7-00

Gelatin - Clay

4% gel sln. on U-coated PET. 120°F. No set, chill set 5 min. (46°F)

\* (No moisture condensed? → Room RH is low?)

ON TABLE TILT

10s chill set equals to No chill set. Because timer starts when the water temp reached 50°F. while plate temp is still pretty high.

2:00pm. gel-7-7-4

4% gel sln. on bare PET. 120°F. No set. chill set starts (46°F)  
on table tilt

3:00pm. gel-7-7-5

4% gel sln. on bare PET. 120°F. No set. chill set 10 min.  
on table tilt.

STILL NO CONDENSATION

4:05pm. STOPPED 5 laponite gel mixture. no bubble. clear.  
total weight: 224.80 g.- C.W. 108.09g

116.7g

4:05pm. gel-7-7-6

4% gel sln. on U-coated PET. 120°F. No set. chill set 10 min.  
on table tilt. No moisture condensation

\* Big difference in Room RH

4:20pm. 5 laponite-7-7-LC-~~17~~ 7

120°F. 1 min. set time. chill set, 10s. on bare PET.

4:25pm. 5 laponite-7-7-LC-6

120°F. 1 min. set time. chill set, 10s. on U-coated PET

## BB 92 83 RESEARCH / DEVELOPMENT

Book No. \_\_\_\_\_

EASTMAN KODAK COMPANY

7-7-00

Item: Gelatin - Clay

1:40 pm. 10 Cloisite gel - 7-7-B-10

105°F coat and kept at 105°F.

5:30 pm. Stop Naugard GPV dispersion (490)

Weight: 461.17 g.

C.W.: 209.78 g.

2.52 g.

7-10-00

STORE ALL Gelatin - containing samples in Freezer ( $\sim -15^{\circ}\text{C}$ )

7-13-00 Collect samples from 7-7. Gelatin - Cloisite film

Peel off: 1. 5 Cloisite gel - 7-7-B-1

2. 5 Cloisite gel - 7-7-B-2

transparent film

3. Cloisite gel - 7-7-B-3

4. Cloisite gel - 7-7-B-4

5. 5 Cloisite gel - 7-7-B-5 on uncoated PET.

(Some sln. was on the back side between PET and gel and very different to peel  $\rightarrow$  (break the film))6. 5 Lapomide gel - 7-7-LC-6 on uncoated PET  
curved.  $\checkmark$ 

7. 5 Lapomide gel - 7-7-LC-7.

8. 10 Cloisite gel - 7-7-B-8

9. 10 Cloisite gel - 7-7-B-9 on uncoated PET

10. 10 Cloisite gel - 7-7-B-10

EXHIBIT C

page 5 of 6 pages

10/633,806

KP 15226-5/86 I. P. S.

Signature \_\_\_\_\_

22 May

The foregoing disclosed to me on

6/1/96

Witness

Date 7-13-00

11. gel - 7-7-1

12. gel - 7-7-2 . on uncoated PET.  
(some sln. in between glasses and PET. difficult to tear )

13. gel - 7-7-3

14. gel - 7-7-4

15. gel - 7-7-5 .

16. gel - 7-7-6 . on uncoated PET

2:00 PM

Samples were conditioned in a 50RH/70°F Room.

1. gel - 7-7-1
2. 10clo3ite - gel - 7-7-B-9 ( on PET )
3. 5Laponitegel - 7-7-LC-7 .
4. 5Clo3itegel - 7-7-B-5
5. 5Clo3itegel - 7-7-B-3
6. 8Laponitegel - 7-7-Le-6
7. 10clo3itegel - 7-7-B-8
8. 5Clo3itegel - 7-7-B-4
9. 5Clo3itegel - 7-7-B-1

EXHIBIT C  
page 6 of 6 pages  
10/633,806

Signature



BB 9283

## RESEARCH / DEVELOPMENT

ebook No. \_\_\_\_\_

EASTMAN KODAK COMPANY

e \_\_\_\_\_ 7-31-00

blem: \_\_\_\_\_

oistiteGel - 7-7-B-2

↓ Chang grips

$$T_1 = 0.7 \text{ mil}$$

\* Using point-flat surface clamp. — before using two flat

$$E = 972269 \text{ psi}, \quad \sigma = 15616, \quad \varepsilon = \text{[redacted]}$$

94090

15114

3.1

29.9

3.7

gel - 7-7-1  $T \sim 1.2 \text{ mil.}$ 

$$E = 565100, \quad \sigma = 14010, \quad \varepsilon = 12.4, \quad \text{Toughness: 125.6.}$$

(71075)

(1056)

(6.7)

5 CloisiteGel - 7-7-B-4

1. ? TD

$$E = 863498, \quad \sigma = 14957, \quad \varepsilon = 4.9, \quad \text{Toughness: 51.6}$$

5 CloisiteGel - 6-27-B-4-2

$$1. \quad \text{TD.} \quad 0.9 \text{ mil, } 836434, \quad 14170, \quad 39.$$

0.8 - 1.0 mil

$$\tilde{E} = 818941, \quad 14537, \quad 6.7, \quad 72.5$$

(48290)

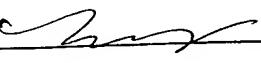
(571)

(3.6)

(45.7)

whitening close to failure edge.

EXHIBIT D

page 1 of 1 page  
10/633,806

Notebook No. \_\_\_\_\_

Date 6-7-00

## Mechanical test of clay-gel

Add test to 5% clay-gel high shear 1

3<sup>4</sup>, 0.85 - 0.50 mil, Input  $\bar{T} = 0.5$  mil. failed inside grips

$$E = 117937 \text{ psi}$$

$$E' = E \times 0.8 = 94370$$

Avg.  $\sigma = 15660$ , 2.3%,  $E = 1220283$ , toughness = 22.6,

Test sample 3% clay-gel high shear 1

(0.9)

Avg.  $T = 0.6$  mil,  $W = 6.35$  mm,  $\epsilon_b = 3.6\%$ ,  $E = 680799$  psi (27603) $\sigma = 14122$  psi (362), toughness = 30.9 (10.6)

Sample 10% clay-gel high shear. Is hard to peel

Thickness measurement

bare film: 3.9 mil

3  $\times$  0.6 - 0.1 mil / thickness

with coating: 4.5 - 4.0

Test of bare PET. 0.189 mil  $\approx$  4.8 mm.Necking  $E = 559876$  $T: 0.65 - 0.35$  mil. Input 0.35 mil $E' = 1635583$ ,  $E = E' \times 0.8 = 1308466$  psi $\sigma = 16076$  psi,  $\epsilon = 0.9\%$  (edge failure)

(True!)

-8-00

Test 1% clay-gel, mechanical test

GL = 2.5", rate = 0.25 /min,  $W = 6.35$  mm,  $\bar{T} = 1.30$  mil  
5 samples $E = 508508$  psi,  $\sigma = 12775$  (425),  $\epsilon = 8.0\%$  (1.1) Toughness = 70.4 (11.8)  
(20698)

## EXHIBIT E

page 1 of 3 pages

10/633,806

Signature \_\_\_\_\_

- 3 -

1/1/01

- 1 -

Pack Freeze-Dry Sample.

The sample is kind of flaky.

1. 10,000 RPM Sediment (6-9-00)

~20.22g.

2. 10,000 RPM LIQUID PART (6-9-00)

~6.89g.

3. 3,000 RPM LIQUID (6-9-00)

(Not Dry enough).

The sample color is not very different from original color

7:15AM Put 2, +3, + residual from the 3000 RPM sediment into a house vacuum oven, raise temp. to 40°C.

9-10AM Peel coating from substrate  
good pencil and pen writability.

Send Tom Blanton two samples for X-ray test

1. 5 dozile gel 615

2. 10 dozile gel 615

Mechanical Test

1. Gel 615, 2. Edge fail, 6). Edge fail

$\overline{W} = 1.5 \text{ ml}$ ,  $\overline{W} = 6.35 \text{ mm}$ .

Ang.  $E = 436843 \text{ (psi)}$ ,  $\epsilon_g = 9.19$ ,  $\sigma_s = 12094 \text{ psi}$ ,  $\sigma_y = 11970 \text{ psi}$   
(12241) (1.1) (650) (719)

Toughness = 77.0 (11.6) ft. lb

EXHIBIT E

page 2 of 3 pages  
10/633,806

problem: Gelatin - Clay (Mechanical Test & Sample Prep.)

2. 5 cloisite gel 615

Avg. 1.03 ml       $\varepsilon_b = 7.4\%$ ,  $E = 671333$  psi,  $\sigma_b = 13553$  psi,  $\sigma_y = 13762$ ,  $T = 71.3$   
 (1.3)                (1942)                (631)                (606)                (72)

3. 10 cloisite gel 615, Brittle failure shear band, whitening

0.83 ml Avg.,  $E = 918043$  psi,  $\varepsilon_b = 6.3$ ,  $\sigma_b = 14457$  psi,  $\sigma_y = 15165$ ,  $T = 67$   
 (45997)                (0.7)                (378)                (703)                (76)

DSC Measurement.

- (1) 10 cloisite gel 615-1,  $w = 15.3$  mg
- (2) 5 cloisite gel 615-1,  $w = 12.7$  mg
- (3) gel 615-1  $w = 17.0$  mg

6-27-00

Prepare Gelatin sln.

7:45AM : (16g gel + 384 g H<sub>2</sub>O20g + 480 g H<sub>2</sub>O)7:50AM Weigh : 20.80g gel + 499.45g H<sub>2</sub>O  $\rightarrow$  520g total  
 $\rightarrow$  (4%) gel soln.

8:15AM

Soak for 20 mins.

Put in 50°C water bath, Mixing using highshear mixer

Weight of small Teflon container: 4108.11 g.

big — — : 320.48 g

Plastic container (400ml): 23.07 g (w). plastic container (1000ml) 55.46 g.

200g total

10g cloisite dispersion (2%) + 190g gel (4%) sln. + 100g H<sub>2</sub>O.

After an hour, take 80g out

EXHIBIT E

page 3 of 3 pages

10/633,806

Notebook No.

5-31-00

Problem: Gelatin - Clay

1:00AM (1) 4% (wt) clay dispersion Laponite RDS from 2 weeks ago

10g clay + 240g water

Clay Na<sup>+</sup> (slight yellow powder)

afterwards - it's like a slurry not transparent in lightening mixer

(2). 4% gelatin solution (30-122)

(3). 10% gelatin solution

(4). (5% clay + gelatin) 1.4%

100g mixture 5g clay + 95g gelatin

+ 50g water (deionized)

Weight of content: 108.09g

total weight: W = 240g

148g

After ~30 mins. 131g. (12g/30 min.)

Need ~90 mins. to evaporate 50g water.

(5) 4% gelatin, 16g gel + 384g H<sub>2</sub>O

Find weight: 211.73g - 108.09g = 103g

Have mixed under high shear for 2.5 hrs.

Clear solution

After 2 hrs. the clay dispersion seems well dispersed

while it's not transparent and has yellow color

! Maybe OK for small amount of addition in gelatin

Took off it from lightening mixer and put in

Stir bar for overnight

EXHIBIT F

page 1 of 1 page  
10/633,806

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